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IS 867 (1963): Methods of test for phenolic moulding materials [PCD 12: Plastics]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 867 - 1963

**[Superseding IS : 867 (Part I) - 1956
and IS : 867 (Part II) - 1959]**

Indian Standard

(Reaffirmed 2003)

**METHODS OF TEST FOR PHENOLIC
MOULDING MATERIALS
(Revised)**

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

Indian Standard

METHODS OF TEST FOR PHENOLIC MOULDING MATERIALS (Revised)

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Indian Standard
METHODS OF TEST FOR PHENOLIC
MOULDING MATERIALS
(Revised)

0. FOREWORD

0.1 This revised Indian Standard was adopted by the Indian Standards Institution on 10 May 1963, after the draft finalized by the Plastics Sectional Committee had been approved by the Chemical Division Council.

0.2 Methods of test for phenolic moulding materials were so far covered by two separate standards, namely, IS : 867 (Part I)-1956 and IS : 867 (Part II)-1959, published in 1956 and 1959 respectively. In these two parts of the standard, methods of sampling of phenolic moulding materials were also given. Recently, it was pointed out that the method of sampling given in the standard was not satisfactory *vis-a-vis* the methods of test prescribed and particularly with the method prescribed for calculation and reporting of test results. It was also suggested that a composite standard laying down methods of sampling of all thermosetting materials would be more useful. The Committee responsible for this standard accepted these suggestions and the methods of sampling have been taken out from IS : 867 (Part I)-1956. The Committee considered it desirable, while making the above changes, to amalgamate the two separate parts of the standard. This revision, therefore, supersedes the two parts, namely, IS : 867 (Part I)-1956 and IS : 867 (Part II)-1959.

0.3 The International Organization for Standardization (ISO) has published the following ISO Recommendations in the field of testing of phenolic moulding materials:

ISO/R 59-1958 PLASTICS. DETERMINATION OF THE PERCENTAGE OF ACETONE SOLUBLE MATTER IN PHENOLIC MOULDINGS

ISO/R 60-1958 PLASTICS. DETERMINATION OF APPARENT DENSITY OF MOULDING MATERIAL THAT CAN BE POURED FROM A SPECIFIED FUNNEL

ISO/R 62-1958 PLASTICS. DETERMINATION OF WATER ABSORPTION

ISO/R 75-1958 PLASTICS. DETERMINATION OF TEMPERATURE OF DEFLECTION UNDER LOAD

ISO/R 119-1959 PLASTICS. DETERMINATION OF FREE PHENOLS IN PHENOL-FORMALDEHYDE MOULDINGS

ISO/R 120-1959 PLASTICS. DETERMINATION OF FREE AMMONIA AND AMMONIUM COMPOUNDS IN PHENOL-FORMALDEHYDE MOULDINGS

ISO/R 171-1961 PLASTICS. DETERMINATION OF BULK FACTOR OF MOULDING MATERIALS

ISO/R 180-1961 PLASTICS. DETERMINATION OF THE IZOD IMPACT RESISTANCE OF RIGID PLASTICS (IZOD IMPACT FLEXURAL TEST)

Necessary changes have been made to align this standard with these ISO Recommendations.

0.4 Wherever a reference to any Indian Standard appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.5 Metric system has been adopted in India and all quantities and dimensions in this standard have been given in this system. Non-metric values to which this industry has been accustomed are also given in brackets, wherever necessary, for the sake of smooth changeover by December 1966.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*).

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for phenolic moulding materials. The tests covered are for determining apparent density, density of moulding and bulk factor, time of flow, shrinkage of moulding, tensile strength, impact strength, cross-breaking strength, water absorption, acetone soluble matter before and after moulding, free phenols, free ammonia and ammonium compounds, temperature of deflection under load, plastic yield, electric strength, surface resistivity after immersion in water, volume resistivity and power factor and permittivity.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Apparent Density — The weight of a unit volume of the loose material under prescribed conditions.

2.2 Bulk Factor — The ratio of the volume of a certain quantity of loose material to its volume after moulding under prescribed conditions.

2.3 Cross-Breaking Strength — The empirical maximum stress in a beam at failure in bend. It is numerically equal to maximum bending moment at failure divided by modulus of section, but the calculated value does not represent the true maximum surface stress as the method of calculation holds good only for an ideal elastic solid.

2.4 Curing Time — In the process of moulding, it is the interval of time from the instant the full pressure is shown on the gauge to the instant the pressure is released.

2.5 Density of Moulding — The weight of a unit volume of the moulded material without inserts.

2.6 Electric Strength — The maximum voltage which the test specimen withstands for a specified period of time without breakdown per millimetre thickness of the specimen.

2.7 Flash Mould — A mould so designed that when charged with an excess of moulding material, the excess escapes at the space in between the relatively moving parts of the mould. This excess or 'flash' sustains part of the total applied pressure.

2.8 Impact Strength — A measure of toughness of a material and is equal to the energy required to break the specimen in one blow.

2.9 Moulding — The shaped article manufactured from moulding material which has been cured by heat and pressure, in a mould. It includes any metal or other accessories inserted during the process of moulding. The moulding may be in the condition in which it was taken from the mould or may have been trimmed, machined or polished subsequently.

2.10 Moulding Material (Phenolic) — The uncured composition, containing thermosetting phenolic synthetic resins, various fillers and other ingredients, which becomes hard, infusible and insoluble (that is, cured) by the application of heat and pressure. The material may be in powder, flake or granular form or these forms compressed to pellets, tablets or preforms.

2.11 Permittivity or Dielectric Constant — The ratio of the capacitance of a capacitor, the dielectric of which consists entirely of the material

under test, to the capacitance of the same configuration of electrodes in vacuum or (practically) in air.

2.12 Plastic Yield — Non-elastic deformation of a beam at a given temperature under a given load.

2.13 Positive Mould — A mould in which the total applied pressure rests only and continuously on the moulding and from which no excess moulding material or flash can escape during the moulding process.

2.14 Power Factor — The ratio of power loss, in watts, of a capacitor, the dielectric of which consists entirely of the material under test, to the applied apparent power, in volt amperes.

2.15 Semi-Positive Mould — A mould which is built on positive lines but which allows a limited amount of escape of the flash as the mould is closed.

2.16 Shrinkage of Moulding — The difference in dimensions, expressed in thousandth of millimetres per millimetre, between a moulding and the mould cavity in which it is moulded, both the mould and the moulding being at normal room temperature when measured.

2.17 Surface Resistivity — The electrical resistance on the surface of the material between the opposite sides of one-centimetre square of the test specimen.

2.18 Synthetic Resin (Phenolic) — A resin produced by the condensation of phenols with aldehydes.

2.19 Tensile Strength — The maximum tensile load sustained by the test specimen under prescribed conditions divided by the original cross-sectional area of the specimen.

2.20 Time of Flow — The time in seconds taken in moulding the material under prescribed conditions.

2.21 Volume Resistivity — The electrical resistance of a unit volume of one-centimetre cube measured between the opposite faces.

3. SAMPLING

3.1 Representative samples of the material shall be drawn as prescribed in IS:2213-1962 Methods of Sampling of Thermosetting Moulding Materials.

4. QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals and distilled water [see IS: 1070-1960 Specification for Water, Distilled Quality (Revised)] shall be employed in tests.

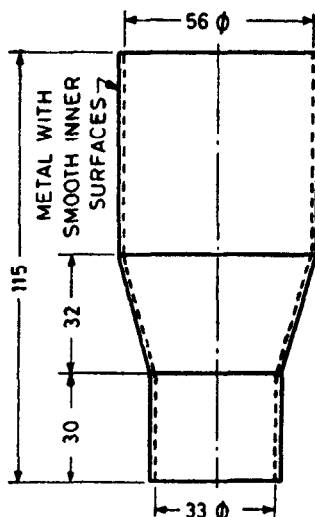
NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

5. DETERMINATION OF APPARENT DENSITY

5.1 Carry out the determination in duplicate.

5.2 Apparatus

5.2.1 *Funnel* — of the shape and dimensions shown in Fig. 1.



All dimensions in millimetres.

FIG. 1 FUNNEL FOR DETERMINING APPARENT DENSITY

5.2.2 *Measuring Cylinder* — may be constructed of metal and having internal diameter 45 ± 5 mm, height 80 ± 5 mm and capacity 100.0 ± 0.5 ml.

5.3 *Procedure* — Mix well the sample of the material before test. Support the funnel vertically with its lower orifice 20 to 30 mm above and coaxial with the measuring cylinder. With the lower orifice of the funnel closed by any convenient means, place a quantity exceeding 100 ml of the material in it. Allow the material to flow into the measuring cylinder. If necessary, assist the flow of the material by loosening it with a rod. When the measuring cylinder is full, draw a blade with a straight edge across the top of the cylinder to remove excess material. Weigh the contents of the measuring cylinder to the nearest 0.1 g.

5.4 *Calculation and Report* — Calculate to two places of decimal the apparent density, that is, the weight in grams of one millilitre of the

material. The mean of the two determinations shall be reported as the representative value of the apparent density of the material.

6. DETERMINATION OF DENSITY OF MOULDING AND BULK FACTOR

6.1 Density of Moulding

6.1.1 Carry out the determination on two test specimens prepared from the same test sample material.

6.1.2 Preparation of Test Specimens — The test specimens shall be in the form of moulded discs with 50.0 ± 1.0 mm (or 2.00 ± 0.04 in) diameter and 3.0 ± 0.2 mm (or 0.125 ± 0.008 in) thick.

6.1.2.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in.²) and a mould temperature of 155° to 170°C. If desired, pre-heat the moulding material before loading into the mould. Do not allow the moulding time to exceed 10 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature, and test within 24 hours of moulding.

6.1.3 Procedure — Attach the test specimen to a piece of wire of sufficient length to reach from the hook of the pan supports to the beaker support so as to suspend about 25 mm above the beaker support and weigh. Weigh the specimen again while immersed with the wire in water at $27^\circ \pm 2^\circ\text{C}$. Make sure that there are no air bubbles on the surface of the specimen and the wire while immersed in water.

6.1.4 Calculation — For the degree of accuracy required, the density of water at 27°C may be taken as 1.0 g/ml. Buoyancy corrections may be neglected and the density of moulding may be calculated as follows:

$$\text{Density of moulding (g/ml) at } 27^\circ\text{C} = \frac{W}{W - w}$$

where

W = weight in g of sample in air, and

w = weight in g of sample in water.

6.1.5 Report — The mean of the two determinations after rounding off to the second place of decimal shall be taken as the representative value of density of moulding for the test sample material.

6.2 Bulk Factor

6.2.1 Calculation — Calculate the bulk factor by dividing the representative value of the density of moulding as determined in 6.1 by the representative value of the apparent density as determined in 5.

7. DETERMINATION OF TIME OF FLOW

7.1 Apparatus — A cup flow mould conforming exactly to the dimensions given in Fig. 2A and 2B.

7.1.1 The mould is standard in the matter of height, diameter, taper, bottom thickness, thermometer-hole positions, polished surface, flash-type construction and flash area.

7.2 Temperature of Test — The temperature of the mould shall be $163^{\circ} \pm 1^{\circ}\text{C}$, the temperature being measured at the inside end of the hole in the body, the bulb of the thermometer being wrapped in copper gauze or metal foil or surrounded by oil to ensure good contact. When not in use, the mould should be kept closed to maintain uniform temperature throughout.

7.3 Pressure — The pressure used on the mould shall be 10.0 ± 0.5 metric tonnes, calculated from the following:

- a) *Up-stroking press* — Ram area $\times$ gauge pressure *minus* weight of ram, platen and mould
- b) *Down-stroking press* — Ram area $\times$ gauge pressure *plus* weight of ram, platen and mould.

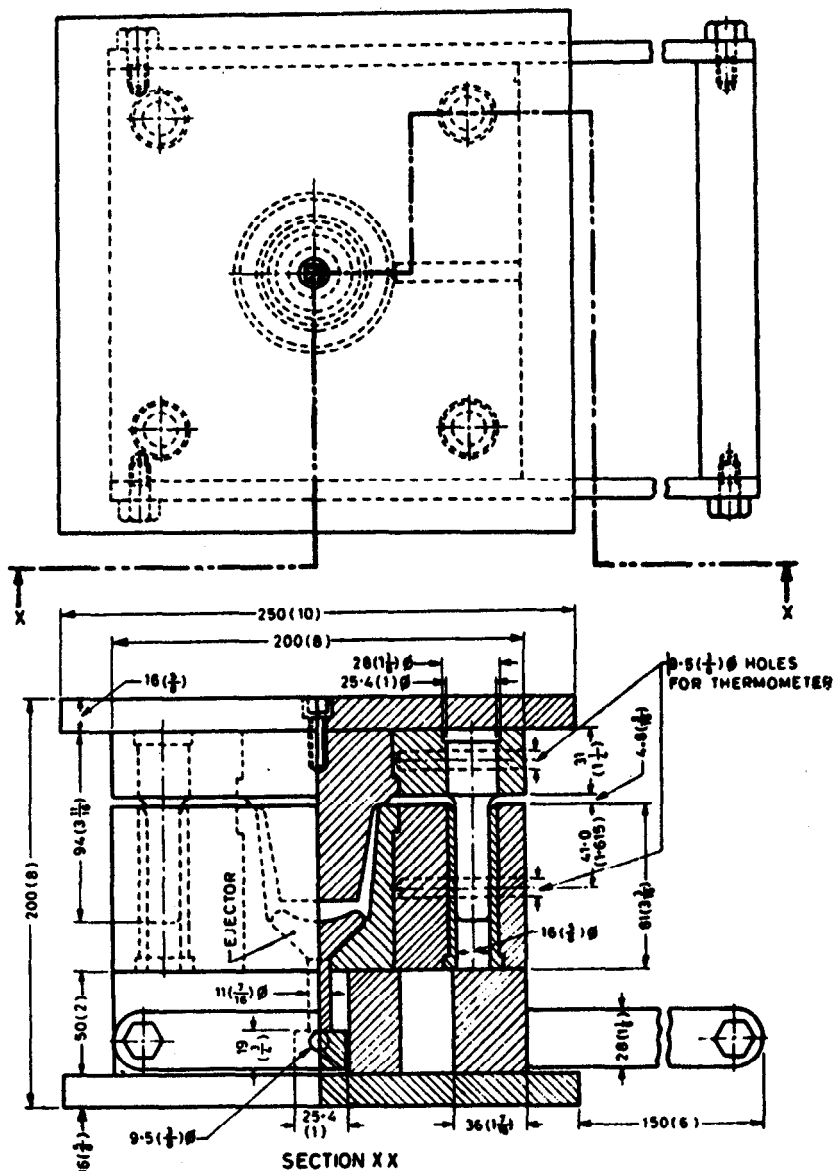
7.3.1 The ram area shall be found from its measured diameter. The pressure gauge shall be checked for accuracy and connected to the press inlet pipe. The line pressure shall not vary by more than ± 3 percent during the course of the test.

7.4 Weight of the Material — The weight of material used shall be sufficient to give a flash of more than 2.0 g and less than 2.5 g in weight.

7.4.1 If a fibrous or other bulky material is to be tested, it shall be compressed before charging so as to form a cylinder of 32 to 38 mm in diameter and not more than 32 mm high.

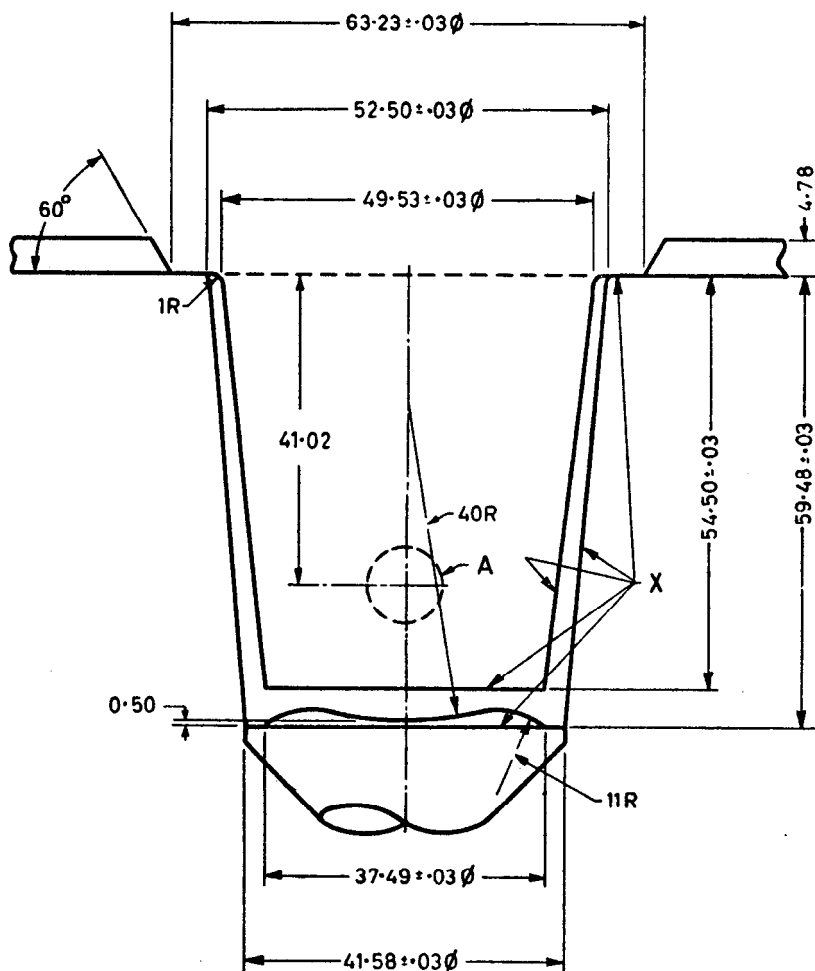
7.5 Procedure

7.5.1 Clean and polish the mould, bring it to the correct temperature and close it. Weigh the material, maintained at room temperature, on to a charging scoop, open the mould sufficient for charging, place the material in the mould and close the mould, the time taken between the dropping of the material into the mould and the closing of the mould



All dimensions in millimetres with inch equivalents in parentheses.

FIG. 2A CUP FLOW MOULD



NOTE — Surfaces marked ' X ' shall be smooth and highly polished. Material to be Mild Steel, case hardened. ' A ' is 9.5 mm dia thermometer hole in body to reach within 12.7 mm of outside wall of moulding.

All dimensions in millimetres.

FIG. 2B DETAILS OF CUP FLOW MOULD

to full pressure on the gauge being between 5 and 10 seconds. Choke the inlet of the hydraulic supply with a bored nipple so that the press ram travels at a speed of 125 mm in 4 to 5 seconds when the valve is fully opened as at the beginning of a test.

7.5.2 Measure the time of flow in seconds on a stop-watch from the instant that the hydraulic gauge shows pressure (indicating that the initial pressure has been applied) to the instant that the flash ceases to move.

7.6 Report — Express the result as 'x seconds flow by the Cup Flow Test', or briefly 'Cup Flow x'.

8. DETERMINATION OF SHRINKAGE OF MOULDING

8.1 Carry out the determination on two test specimens prepared from the same test sample material.

8.2 Preparation of Test Specimens — The test specimens shall be in the form of moulded discs 100 to 125 mm (or 4.0 to 5.0 in.) in diameter and 3.0 ± 0.2 mm (or 0.125 ± 0.010 in.) thick, and shall be flat.

8.2.1 Mould the specimens in a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in.²) and a mould temperature of $155^\circ \pm 1^\circ\text{C}$. Do not pre-heat the moulding material before loading into the mould and do not allow the mould to breathe. Mould the specimens for 4.00 ± 0.25 minutes, eject the mouldings while hot and allow to cool in air to $27^\circ \pm 2^\circ\text{C}$ and measure the dimensions.

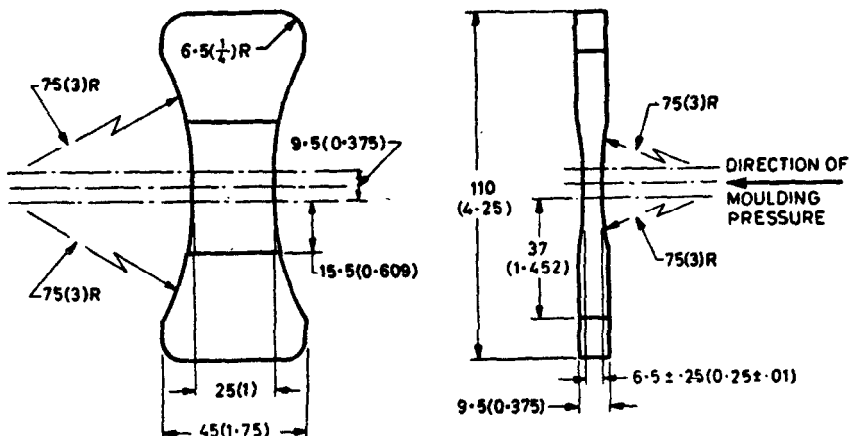
8.3 Calculation — Calculate the shrinkage of moulding by subtracting the diameter of the cold moulding from that of the cold mould and divide by the diameter of the cold mould, the measurements being made on a marked diameter to avoid possible inaccuracy due to the specimen not being perfectly circular. Express the shrinkage in thousandths of a millimetre per millimetre (or mils per inch).

8.4 Report — The mean of the two determinations shall be taken as the representative value of shrinkage of moulding for the test sample material.

9. DETERMINATION OF TENSILE STRENGTH

9.1 Carry out the determination on three test specimens prepared from the same test sample material.

9.2 Preparation of Test Specimens — The test specimens shall conform to dimensions shown in Fig. 3.



All dimensions in millimetres with inch equivalents in parentheses.

FIG. 3 SPECIMEN FOR TENSILE TEST

9.2.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in.²) and a mould temperature of 155° to 170°C, the direction of moulding pressure being as shown in Fig. 3. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed 15 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature. Note that all surfaces of the specimens are free from visible flaws, scratches or imperfections. Condition the specimens as prescribed in 9.3 and test within 24 hours of moulding.

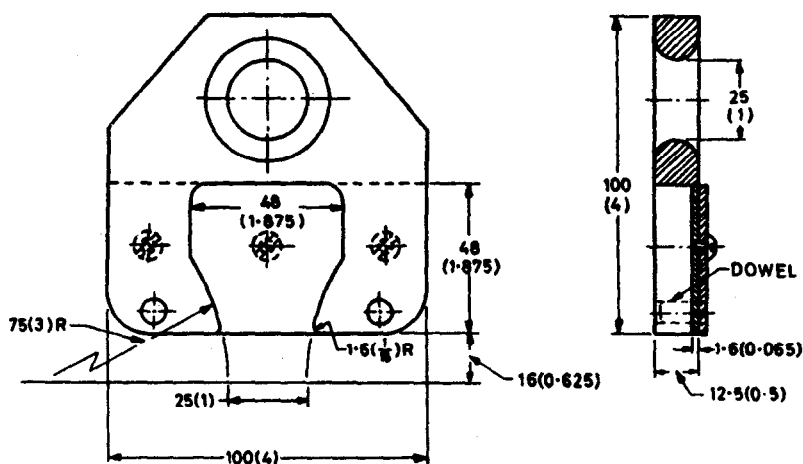
9.3 Conditioning — Keep the test specimens in a desiccator over anhydrous calcium chloride for at least 16 hours before testing.

9.3.1 Special conditioning procedure may be adopted if so agreed to between the purchaser and the supplier.

9.3.2 Conditioning may not be done for routine plant control, but in case of dispute, the specimens shall be conditioned as prescribed in 9.3 or 9.3.1.

9.4 Procedure — Allow a definite time interval, not exceeding 60 minutes, to elapse between removal of the specimen from the desiccator and application of load. Measure the width and thickness of the specimen at several places near the centre of the specimen. Use any standard tensile testing machine provided it is possible to measure the load at fracture to within one percent. Hold the specimen even

and firmly in grips as shown in Fig. 4. Increase the load evenly so that the specimen breaks within 0.5 to 1.5 minutes from the start of the loading. Record the maximum load carried by the specimen during the test.



All dimensions in millimetres with inch equivalents in parentheses.

FIG. 4 HOLDER FOR TENSILE TEST SPECIMEN

9.5 Calculation — Express the tensile strength of each specimen in kg/cm^2 (or lb/in^2) of the original minimum cross-sectional area calculated from the measured dimensions of the specimen.

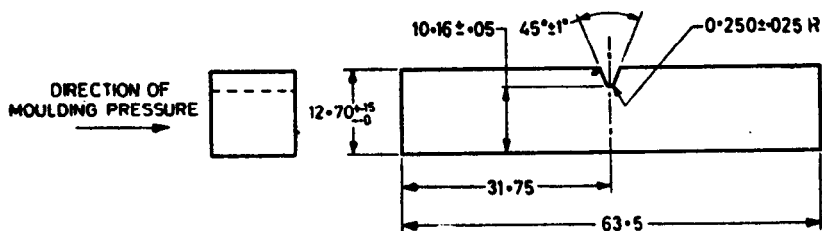
9.6 Report — The mean of the three determinations shall be taken as the representative value of tensile strength for the test sample material.

10. DETERMINATION OF IMPACT STRENGTH

10.1 Carry out the determination on at least five test specimens prepared from the same test sample material.

10.2 Preparation of Test Specimens — The moulded (or moulded and then machined) specimens shall conform to dimensions shown in Fig. 5, except that the notched face and the face opposite to it shall be moulded surfaces.

10.2.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm^2 (or 1 to 3 ton/in^2) and a mould temperature of 155° to 170°C, the direction of moulding pressure being as shown in Fig. 5. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed



All dimensions in millimetres.

FIG. 5 SPECIMEN FOR IMPACT TEST

15 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature. Condition the specimens as prescribed in 9.3 and test within 24 hours of moulding.

10.3 Procedure

10.3.1 Accurately mount the specimen in the vice of a pendulum (Izod) type impact testing machine as shown in Fig. 6, with the notch facing the striker and the root of the notch level with the horizontal face of the vice. Strike the specimen at a distance of 22.0 ± 0.5 mm (or 0.866 ± 0.020 in) from the face of the vice, the velocity of the striking edge at the moment of impact being 244 ± 3 cm (or 8.0 ± 0.1 ft) per second.

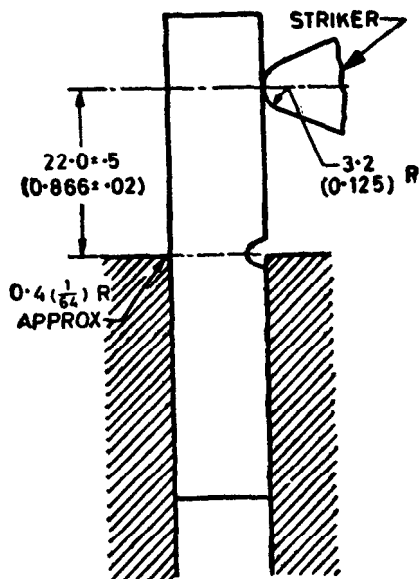
10.3.2 The gripping edge of the vice shall have a radius of approximately 0.4 mm (or $1/64$ in). The pendulum shall be so constructed that the centre of percussion coincides with the centre of the striking edge. This edge shall have a radius of 3.2 mm (or $1/8$ in), and shall strike squarely across the full width of the specimen.

10.3.3 Test specimens of impact strength not exceeding 0.12 kg·m (or 0.85 ft·lb) shall be tested in an Izod pendulum machine with a striking energy of 0.14 kg·m (or 1.0 ft·lb). Test specimens of impact strength exceeding 0.12 kg·m (or 0.85 ft·lb), but not exceeding 0.35 kg·m (or 2.5 ft·lb), shall be tested in an Izod pendulum machine with a striking energy of 0.42 kg·m (or 3.0 ft·lb).

10.4 Report — Express the impact strength of each specimen in kg·m (or ft·lb) of energy absorbed in breaking the specimen. The mean of the five determinations shall be taken as the representative value of impact strength of the test sample material.

11. DETERMINATION OF CROSS-BREAKING STRENGTH

11.1 Carry out the determination on three test specimens prepared from the same test sample material.



All dimensions in millimetres with inch equivalents in parentheses.

FIG. 6 SET UP FOR IMPACT TEST SPECIMEN

11.2 Preparation of Test Specimens — The specimens shall consist of moulded rectangular bars of minimum length 115 mm (or 4.5 in), width 12.7 ± 0.2 mm (or 0.500 ± 0.010 in) and thickness 9.50 ± 0.25 mm (or 0.375 ± 0.010 in).

11.2.1 Mould the specimens in a flash, a positive or a semi-positive mould with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²) and a mould temperature of 155° to 170°C. Apply the moulding pressure in a direction perpendicular to the largest face of the specimens. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed 8 minutes, but a shorter time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature. Condition the specimens as prescribed in 9.3 and test within 24 hours of moulding.

11.3 Procedure — Determine the mean width and the mean thickness of each specimen. Place the specimen symmetrically across parallel V-shaped supports, the distance between the supporting edges being 100.0 to 102.0 mm. Apply a load squarely across the width of the specimen by means of a third V-shaped block parallel to and midway between the supporting blocks. The contact edges of the supporting

blocks and the block applying the load shall have a radius of approximately 1.6 mm (or 1/16 in). Increase the load steadily so that the specimen fractures in 15 to 45 seconds. Note the load in kilograms at failure.

11.4 Calculation

$$\text{Cross-breaking strength (kg/cm}^2\text{)} = \frac{1.5WL}{BD^2}$$

where

W = load in kg at failure,

L = distance in cm between supports,

B = mean width in cm of specimen, and

D = mean thickness in cm of specimen.

11.5 Report — The mean of the three determinations shall be taken as the representative value of the cross-breaking strength of the test sample material.

12. DETERMINATION OF WATER ABSORPTION

12.1 Carry out the determination on three test specimens prepared from the same test sample material.

12.2 Preparation of Test Specimens — The specimens shall be in the form of moulded discs 50.0 ± 1.0 mm (or 2.00 ± 0.04 in) in diameter and 3.0 ± 0.2 mm (or 0.125 ± 0.010 in) thick.

NOTE — Comparison of water absorption of materials is possible only when test specimens of identical dimensions are used.

12.2.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²) and a mould temperature of 155° to 170°C. If desired, pre-heat the moulding material before loading into the mould. Do not allow the moulding time to exceed 15 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature and test within 24 hours of moulding.

12.3 Procedure — Dry the specimens in an oven for 24 ± 1 hours at $50^\circ \pm 2^\circ\text{C}$, cool in a desiccator and weigh each specimen to the nearest milligram (W_1). Immediately place them in a container of water maintained at $27.0^\circ \pm 1.0^\circ\text{C}$. Take precautions to prevent the specimens from making contact over any substantial area with one another or with the container. Take the specimens away from the water after 24 ± 1 hours and remove all surface water with a clean dry cloth or with filter

paper. Re-weigh the specimens within one minute of taking them out from the water (W_2).

12.3.1 When it is desired to allow for the presence of water-soluble matter, re-dry the specimens in an oven for 24 ± 1 hours at $50^\circ \pm 2^\circ\text{C}$ after completing the procedure prescribed in 12.3. Cool the re-dried specimens in a desiccator and re-weigh (W_3).

12.4 Calculation — The water absorption of a test specimen is the difference in milligrams between the weight before immersion and the weight after immersion, that is ($W_2 - W_1$). The mean of the three determinations shall be taken as the representative value of water absorption for the test sample material.

12.4.1 When allowing for the presence of water-soluble matter, the water absorption of a test specimen is the difference in milligrams between the weight after immersion and the weight after re-drying, that is ($W_3 - W_1$).

12.5 Report — The mean of the three determinations shall be taken as the representative value of water absorption for the test sample material.

13. DETERMINATION OF ACETONE SOLUBLE MATTER BEFORE MOULDING

13.1 Carry out the determination in duplicate.

13.2 Apparatus

13.2.1 Extraction Apparatus — of the type shown in Fig. 7. Alternatively, a Soxhlet type apparatus in which the material in the thimble is surrounded by the vapour of the boiling solvent may be used. Any other extraction apparatus may be used provided it gives similar results.

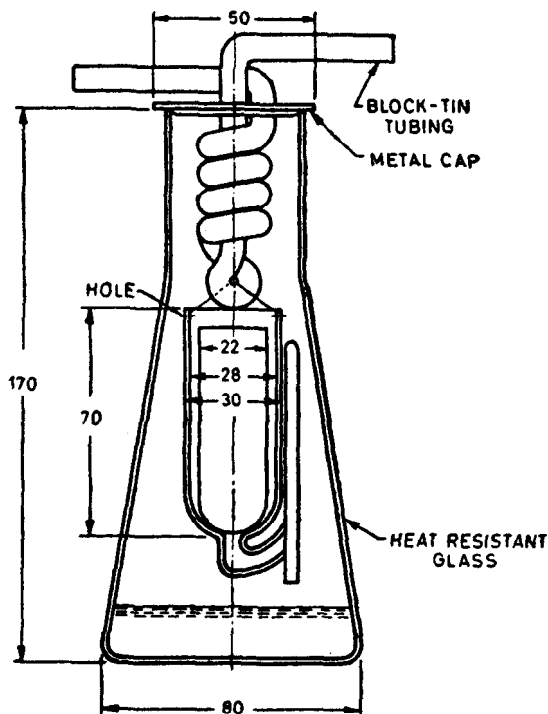
13.3 Reagent

13.3.1 Acetone — conforming to *IS: 170-1950 Specification for Acetone.

13.4 Preparation of Sample — If the material is in the form of preforms, flakes, coarse pieces or sheet, reduce it to a powder (see Note) or small pieces before test, taking care that no overheating of the material occurs during the breaking process. Dry about 4 to 5 g of the material at room temperature in vacuum over concentrated sulphuric acid or any other desiccant for 24 hours.

NOTE — The material shall not be ground to a very fine powder which may tend to agglomerate in the thimble.

*Since revised.



All dimensions in millimetres.

FIG. 7 EXTRACTION APPARATUS

13.5 Procedure — Dry a single thickness extraction thimble for two hours at $105^{\circ} \pm 2^{\circ}\text{C}$ and cool it in a desiccator and weigh. Place about 3 g of the sample in the extraction thimble and weigh again. Close the thimble with a loose plug of absorbent cotton wool so that none of the material may float out. Weigh the thimble and contents together with the loose plug of absorbent cotton wool. Place it in the siphon of the extraction apparatus. Fit the condenser, siphon tube and flask together and add 100 ml of acetone. Regulate the heating so that siphoning takes place at the rate of 20 to 30 times per hour. Continue the extraction for 16 hours. At the end of this time, dry the thimble and contents together at room temperature in vacuum over concentrated sulphuric acid or other desiccant for 24 hours and weigh. All the weighings shall be done to the nearest 0.001 g.

13.6 Calculation

$$\text{Acetone soluble matter before moulding, percent by weight} = \frac{W - w}{W} \times 100$$

where

W = weight in g of the sample taken for the test, and

w = weight in g of the residue obtained after extraction.

13.7 Report — The mean of the two determinations shall be taken as the representative value of acetone soluble matter before moulding for the test sample material.

14. DETERMINATION OF ACETONE SOLUBLE MATTER AFTER MOULDING

14.1 Carry out the determination in duplicate.

14.2 Reagent

14.2.1 Acetone — conforming to*IS: 170-1950 Specification for Acetone.

14.3 Preparation of Sample — Obtain drillings, filings, shavings or the like from a moulding prepared as prescribed in 8.2 and 8.2.1 except that the moulding of the specimen shall be done for 8 minutes, taking care that during this breaking process no undue heating of the material occurs. Avoid contamination of the material with foreign matter. Take the portion of the material which passes through a 425-micron IS Sieve but is retained on a 250-micron IS Sieve. Immediately after sieving, place the ground material in an air-tight container to prevent any absorption of moisture.

14.4 Procedure — Weigh to the nearest milligram about 3 g of the sample into a tared open-texture quantitative filter paper or a single thickness extraction thimble. Fold over the thimble or filter paper containing the sample in such a manner that none of the material may float out. Place it in the siphon of any of the extraction apparatus specified in 13.2. Fit the condenser, siphon tube and flask together and add 50 ml of acetone. Regulate the heating so that siphoning takes place at the rate of 20 to 30 times per hour. Continue the extraction for 6 hours. At the end of this time, remove the flask and pour the acetone extract into a dish or into a smaller flask which has been weighed to the nearest milligram. Wash the flask from which the contents have been poured with about 20 ml of acetone and add the washings to the extract. Evaporate the acetone by any convenient means, care being taken that the temperature does not exceed 50°C. Place the dish or flask containing the residue in a well-ventilated oven at $50^\circ \pm 2^\circ\text{C}$ for 30 minutes, cool in

*Since revised.

a desiccator and weigh. Repeat the process of heating, cooling and weighing until the weight is constant to within 3 milligrams.

14.5 Calculation

$$\text{Acetone soluble matter after moulding, percent by weight} = 100 \frac{w}{W}$$

where

w = weight in g of the residue obtained, and

W = weight in g of the sample taken for the test.

14.6 Report — The mean of the two determinations shall be taken as the representative value of acetone soluble matter after moulding of the test sample material.

15. DETERMINATION OF FREE PHENOLS

15.1 Carry out the determination in duplicate.

15.2 Reagents

15.2.1 Iodine Solution — approximately N/20, prepared by dissolving 6.35 g of iodine in approximately 40 ml of a 50 percent solution of potassium iodide and diluting to 1 000 ml with water.

15.2.2 Sodium Tetraborate

15.2.3 Dilute Sulphuric Acid — approximately 2 N.

15.2.4 Standard Sodium Thiosulphate Solution — N/20, freshly standardized.

15.2.5 Starch Indicator Solution — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

15.3 Preparation of Sample — Reduce to powder form a specimen prepared as prescribed in 8.2 and 8.2.1, taking care that no overheating of the material occurs during the breaking process. Avoid contamination of the material with foreign matter. Take the portion of the material which passes through a 250-micron IS Sieve. Immediately after sieving, place the sample in an air-tight container until required for use.

15.4 Procedure — Weigh to the nearest 0.01 g about 5 to 10 g of the sieved sample, place in a 250-ml glass-stoppered flask and cover with ten times its weight of water heated to 90° to 100°C. Stopper the flask and shake it so that the material is thoroughly wetted. Allow it to cool

at room temperature for one hour with occasional shaking. Filter the contents of the flask through a sintered glass crucible of medium porosity. Place 5 ml of the aqueous extract in a 250-ml iodine flask. Add 10 ml of iodine solution and 3 to 4 g of sodium tetraborate and dilute with water to about 100 ml. Allow the mixture to stand for ten minutes, and then add 20 to 30 ml of dilute sulphuric acid. Titrate the excess iodine against standard sodium thiosulphate solution using 2 ml of starch solution as indicator.

15.4.1 Carry out a blank determination, using 5 ml of water instead of the aqueous extract.

15.5 Calculation

Free phenols (as C_6H_5OH), percent by weight = $0.157 (V_1 - V_2)$
where

V_1 = volume in ml of standard sodium thiosulphate solution used in the blank titration, and

V_2 = volume in ml of standard sodium thiosulphate solution used in titration of the aqueous extract.

15.6 Report — The mean of the two determinations shall be taken as the representative value of free phenols for the test sample material.

16. DETERMINATION OF FREE AMMONIA AND AMMONIUM COMPOUNDS

16.1 Carry out the determination in duplicate.

16.2 Reagents

16.2.1 *Water* — free from ammonia.

16.2.2 *Potassium Permanganate* — solid.

16.2.3 *Sodium Hydroxide Solution* — 2 percent.

16.2.4 *Nessler Reagent* — Dissolve 10 g of potassium iodide in 10 ml of water and add to it slowly, with stirring, a saturated aqueous solution of mercuric chloride until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and, when it has dissolved, add one millilitre more of the mercuric chloride solution and dilute to 200 ml with water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

16.3 Preparation of Sample — Reduce to powder form a specimen prepared as prescribed in 8.2 and 8.2.1, taking care that no overheating of the material occurs during the breaking process. Avoid contamination

of the material with foreign matter. Take the portion of the material which passes through a 250-micron IS Sieve. Immediately after sieving, place the sample in an air-tight container until required for use. The extraction with water shall be carried out within one hour of grinding the moulding.

16.4 Procedure — Weigh to the nearest 0.01 g about 5 to 10 g of the sieved sample, place in a 250-ml glass-stoppered flask and cover with ten times its weight of water heated to 90° to 100°C. Stopper the flask and shake it so that the material is thoroughly wetted. Allow it to cool at room temperature for one hour with occasional shaking. Filter the contents of the flask without using suction. Pipette 10 ml of the filtrate into a 250-ml distillation flask (*see* Note 1) and add 0.1 g of potassium permanganate and 10 ml of sodium hydroxide solution. Distil the mixture slowly and collect the first 15 ml of the distillate in a 50-ml Nessler's tube. Dilute this to 50 ml with water, add 2 ml of Nessler reagent, and determine the percentage of ammonia by matching the colour of the diluted distillate with that of a number of standard solutions of known ammonia content (*see* Note 2). The Nessler reagent should be added to the diluted distillate and to the standard solutions at the same time.

NOTE 1 — Before using the distillation apparatus, it shall be freed from any ammonia, which may be present, by distilling a quantity of water in it until the addition of 2 ml of Nessler reagent to 50 ml of distillate does not give rise to any colour.

NOTE 2 — If the distillate is found to contain more than 0.06 mg of ammonia, a 10-ml portion of the filtrate shall be suitably diluted and a 10-ml portion of this diluted solution shall be used in the distillation.

16.4.1 Alternatively, calibrated glass colour discs may be used for the determination of free ammonia and ammonium compounds provided the instructions of the manufacturer are followed, but in case of dispute, test method prescribed in 16.4 shall be followed.

16.5 Calculation

$$\begin{array}{l} \text{Free ammonia and ammonium compounds} \\ \text{(as NH}_3\text{), percent by weight} \end{array} = \frac{SD}{10}$$

where

S = number of mg of ammonia in the matching standard solution; and

D = dilution factor, if the filtrate is diluted before distillation.

16.6 Report — The mean of the two determinations shall be taken as the representative value of free ammonia and ammonium compounds for the test sample material.

17. DETERMINATION OF TEMPERATURE OF DEFLECTION UNDER LOAD

17.1 Carry out the determination on two test specimens, prepared from the same test sample material.

17.2 Apparatus — The apparatus shall be assembled as shown in Fig. 8.

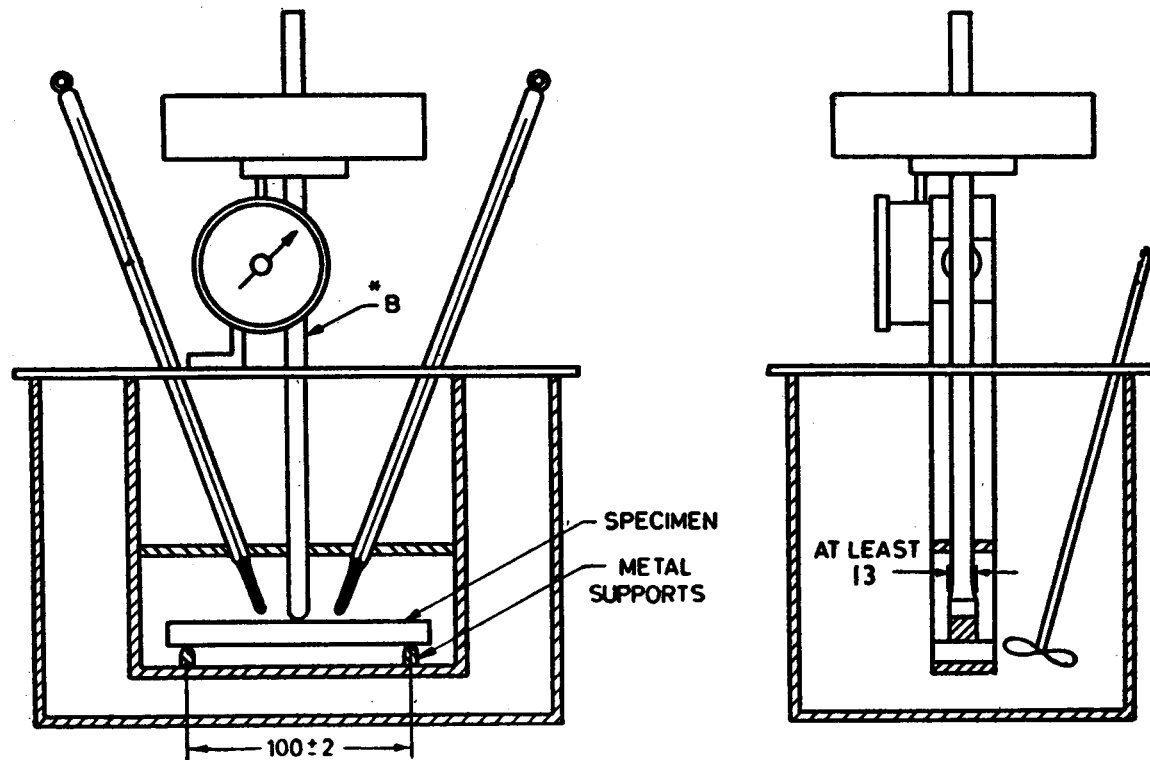
17.2.1 Specimen Supports — The specimen shall be supported on metal supports, 100 ± 2 mm apart, with the load applied on top of the specimen vertically and midway between the supports. The contact edges of the supports and of the rod by which pressure is applied shall be rounded to a radius of 3.0 ± 0.2 mm. The vertical members which attach the specimen supports to the upper plate shall be made of material having the same coefficient of linear expansion as of the rod by which the load is applied.

NOTE — Unless these parts have the same coefficient of linear expansion, the differential change in length of these parts introduces an error in the reading of the apparent deformation of the specimen. A test should be made on each apparatus using a test bar made of materials having a low coefficient of expansion (Invar or borosilicate glass have been found suitable for this purpose). The temperature range to be used should be covered and a correction factor determined for each temperature. If this factor is 0.010 mm or greater, its algebraic sign should be noted and the factor should be applied to each test by adding it algebraically to the reading of apparent deflection of the test specimen.

17.2.2 Immersion Bath — containing a suitable liquid heat transfer medium (see Note) in which the specimens shall be immersed. It shall be kept well stirred during the test and shall be provided with suitable means for raising the temperature at an average rate of 2°C per minute and the temperature shall not deviate from the average by more than $\pm 1^{\circ}\text{C}$ at any time.

NOTE — A liquid heat transfer medium shall be chosen which is stable at the temperatures used, and also which does not affect the specimen at these temperatures. Mineral oil is found to be suitable.

17.2.3 Weights — a set of weights of suitable sizes so that the specimens may be loaded to a fibre stress either of $18.5 \text{ kg/cm}^2 \pm 2.5$ percent for Method I or $4.6 \text{ kg/cm}^2 \pm 2.5$ percent for Method II. The weight of the rod which applies the testing force shall be determined and included as part of the total load. If a dial gauge is used, the force exerted by its spring shall be determined and shall be included as part of the total load (see Notes 1 and 2). The load shall be calculated from



*B is a metal rod through which load is applied and is made of material of same coefficient of linear expansion as that of the vertical members which attach the metal supports to the upper plate. The contact edges shall be rounded to a radius of 3.0 ± 0.2 mm.

All dimensions in millimetres.

FIG. 8 APPARATUS FOR DETERMINATION OF TEMPERATURE OF DEFLECTION UNDER LOAD

the following formula:

$$P = \frac{2 S b d^3}{3 w}$$

where

P = load in kg;

S = extreme fibre stress in specimen (18.5 kg/cm² when tested according to Method I, and 4.6 kg/cm² when tested according to Method II);

b = width in cm of the specimen;

d = thickness in cm of the specimen; and

w = width in cm of span between supports.

The dimensions b , d and w shall be measured to the nearest 0.1 mm.

NOTE 1 — In some designs of this apparatus, the force of the dial-gauge spring is directed upwards and shall be subtracted from the load, while in other designs, this force acts downwards and shall be added to the load.

NOTE 2 — Since the force exerted by the spring in certain dial gauges varies considerably over the stroke, this force should be measured in that part of the stroke which is to be used.

17.2.4 Thermometers — The thermometers shall be mercury-in-glass thermometers of the partial immersion type, calibrated in degrees centigrade. Graduation marks shall be spaced at 1°C intervals or closer, and the scale-error at any reading shall not exceed 0.5°C. The thermometers shall be immersed to the depth to which they have been calibrated and which shall not be less than 50 mm.

17.2.5 Preparation of the Apparatus — The apparatus shall be arranged so that the deflection of the midpoint of the specimen is measured on a scale calibrated in hundredths of a millimetre. The apparatus may be arranged to shut off the heat automatically and sound an alarm when the specified deflection has been reached.

17.3 Preparation of Test Specimens — The specimens, at least 110 mm in length, 3.0 to 4.2 mm in width and 9.8 to 12.8 mm in thickness, shall be moulded in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²), the mould temperature being 155° to 170°C. If desired, pre-heat the material before loading into the mould. Do not allow curing time to exceed 10 minutes; but a shorter time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings.

17.4 Conditioning — Keep the test specimens at 27° ± 2°C and 65 ± 5 percent relative humidity for 90 ± 2 hours.

17.5 Method I

17.5.1 Procedure — Place the test specimen in the apparatus with its thickness in a vertical plane, in such a manner that the direction of the testing force is perpendicular to the direction of the moulding pressure. Place the thermometers so that they extend to within 2 mm of the specimen but do not touch it. Maintain the bath temperature at $27^{\circ} \pm 1^{\circ} \text{C}$ at the start of each test. Adjust the load so that fibre stress is $18.5 \text{ kg/cm}^2 \pm 2.5$ percent as calculated by the formula given in 17.2.3. Allow the load to act for 5 minutes (*see Note*). Note the zero reading or setting of the measuring device and start the heating. Conduct the test by raising the temperature of the bath as prescribed in 17.2.2. Note the temperature at which the test specimen has deflected through the standard deflection corresponding to the thickness of the test specimen, as indicated in 17.5.2. The mean of the two determinations shall be taken as the representative value of temperature of deflection under a load of 18.5 kg/cm^2 fibre stress.

NOTE — The 5-minute waiting period is provided to compensate particularly for the creep exhibited by several materials at room temperature when subjected to the prescribed fibre stress. That part of the creep which occurs in the initial 5 minutes is usually a large fraction of that which occurs in the first 30 minutes.

17.5.2 Standard Deflection versus Thickness of Test Specimen

THICKNESS OF TEST SPECIMEN	STANDARD DEFLECTION
mm	mm
9.8	0.33
10.0	0.32
11.0	0.29
12.0	0.27
12.8	0.25

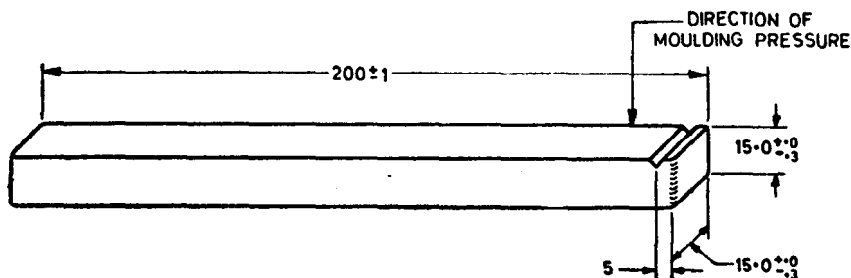
17.6 Method II

17.6.1 Procedure — Conduct the test exactly as prescribed under 17.5 except that the load shall be adjusted to give a fibre stress of $4.6 \text{ kg/cm}^2 \pm 2.5$ percent as calculated by the formula given under 17.2.3. Note the temperature at which the test specimen has deflected through the standard deflection corresponding to the thickness of the test specimen as prescribed under 17.5.2. The mean of the two determinations shall be taken as the representative value of temperature of deflection under a load of 4.6 kg/cm^2 fibre stress.

18. DETERMINATION OF PLASTIC YIELD

18.1 Carry out the determination on two test specimens prepared from the same test sample material.

18.2 Preparation of Test Specimens — The test specimens shall conform to the dimensions shown in Fig. 9.



All dimensions in millimetres.

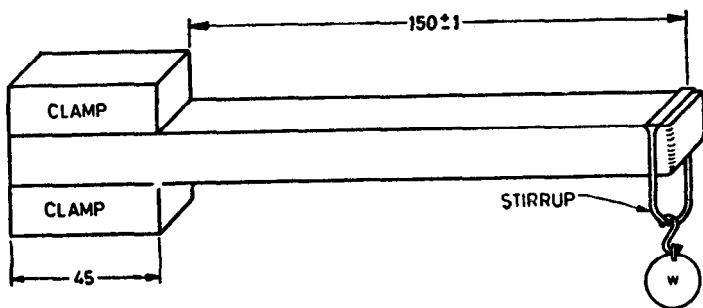
FIG. 9 SPECIMEN FOR PLASTIC YIELD TEST

18.2.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²) and a mould temperature of 155° to 170°C, the direction of moulding pressure being as shown in Fig. 9. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed 15 minutes; but a shorter curing time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the moulding. Cool the specimens to room temperature and test within 24 hours of moulding.

18.3 Procedure

18.3.1 Carry out the test in an oven maintained within $\pm 2^\circ\text{C}$ of the temperature specified in the individual standard for the moulding material. Fix the specimen in a rigid manner in the oven by means of a clamp as shown in Fig. 10, and suspend a suitable stirrup with a rigid knife-edge, carrying an attachment for supporting the weight from the notch in the unsupported end of the specimen. Do not allow the weight of the stirrup and attachment to exceed 20 g.

18.3.2 Fifteen minutes after placing the specimen in the oven, measure the height of the unsupported end of the specimen with reference to a suitable datum point in millimetres to an accuracy of 0.1 mm in such a manner that the temperature of the oven does not fall below the specified limit while the measurement is being taken. Then attach a weight to the stirrup so that the total load, including the stirrup, applied to the specimen is 450 ± 1 g. Maintain the specimen in the oven under these



All dimensions in millimetres.

FIG. 10 ARRANGEMENT OF SPECIMEN FOR PLASTIC YIELD TEST

conditions for 6 hours $\pm$ 10 minutes. At the end of this period, again measure the height of the unsupported end of the specimen, with reference to the datum point, while the specimen is still under the test conditions of load and temperature.

18.4 Calculation — Record the difference between the initial and the final measurements and express in millimetres as the plastic yield of the specimen at the specified temperature.

18.5 Report — The mean of the two determinations shall be taken as the representative value of plastic yield for the test sample material.

19. DETERMINATION OF ELECTRIC STRENGTH

19.1 Carry out the determination on at least four test specimens prepared from the same test sample material. One of these specimens shall be used only to provide a preliminary estimate of the approximate value of the voltage at which the test specimens are likely to fail.

19.2 Apparatus

19.2.1 Electrodes — The bottom electrode shall consist of a solid cylinder of brass 80 mm (or 3 in) in diameter and 25 mm (or one inch) in thickness. The upper electrode shall consist of a solid cylinder of brass 40 mm (or 1.5 in) in diameter and 40 mm (or 1.5 in) in thickness. The sharp edges of the electrodes shall be rounded to a radius not exceeding one millimetre (or 0.04 in). The test faces shall be kept smooth and polished and free from pitting. The electrodes shall be held truly coaxial while testing.

19.2.2 Testing Transformer—Alternating voltage, approximately of the sine wave form and of 50 cycles per second frequency, shall be used. The wave form of the test voltage shall not vary by more than ± 5 per cent from a pure sine wave (that is, the crest factor shall be within 1.343 to 1.484). The rms value of the applied voltage shall be determined from the readings of a peak voltmeter suitably connected to the output side of the transformer. For this purpose, the wave form shall be deemed to be sinusoidal. (The rms value may also be determined by an electrostatic voltmeter connected to the output side or even by an ordinary ac voltmeter connected to the input side or to a tertiary coil, provided that this voltmeter has been properly calibrated against a sphere gap connected to the output side.) The capacity of the transformer shall be sufficient to maintain the full test voltage on the specimen for the maximum time required for the test. A rating of 2 to 5 kVA depending upon the output voltage shall normally be considered sufficient. The transformer circuit should be protected from excessive current by a circuit-breaker on breakdown of the test specimen.

19.2.3 Voltage Control—Control of voltage may be secured by using one of the following devices:

- a) Variable ratio auto-transformer,
- b) Induction regulator,
- c) Generator field regulator (for separate alternator), and
- d) Resistance potential divider.

19.3 Preparation of Test Specimens—The specimens shall be moulded discs not less than 100 mm (or 4.00 in.) in diameter and 3.0 ± 0.2 mm (or 0.125 ± 0.010 in.) thick. The specimens shall be flat.

19.3.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in.²) and a mould temperature of 155° to 170°C. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed 6 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature and test within 24 hours of moulding.

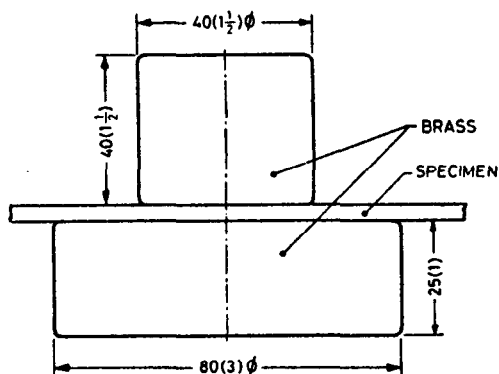
19.4 Conditioning—Keep the specimens for at least 5 hours in a desiccator over anhydrous calcium chloride at room temperature prior to testing.

19.5 Procedure

19.5.1 Immerse the four specimens and the electrodes for 15 to 25 minutes in a suitable bath of insulating oil [conforming to *IS: 335-1953

*Since revised.

Specification for Insulating Oil for Transformers and Switchgear (Low Viscosity Type)] maintained at $90^{\circ} \pm 2^{\circ}\text{C}$. At the end of this time, apply a voltage to one specimen mounted symmetrically between the electrodes as shown in Fig. 11 and increase it uniformly at about 30kV per minute until breakdown occurs. Treat this as a preliminary estimate of the approximate value of the voltage at which other specimens are likely to fail. Mount the remaining 3 specimens in turn between the electrodes and apply a voltage equal approximately to half the breakdown voltage of the first specimen and maintain for 20 seconds. If the specimen withstands this voltage without failure, raise the test voltage in steps of 1 kV up to 25 kV and thereafter in steps of 2 kV.



All dimensions in millimetres with inch equivalents in parentheses.

FIG. 11 ARRANGEMENT OF ELECTRODES FOR ELECTRIC STRENGTH TEST

19.5.2 Maintain each test voltage for 20 seconds unless failure occurs. If the second or any subsequent specimens fail in less than 20 seconds after the commencement of testing, disregard the test and repeat it on a fresh specimen, the initially applied voltage stress being approximately one fourth of the breakdown voltage of the first specimen in this case.

19.6 Calculation — The electric strength of the specimen shall be the maximum voltage which the specimen withstands for 20 seconds without breakdown, divided by the thickness of the specimen in millimetres (or thousandths of an inch) measured near the point of breakdown.

19.7 Report — The mean of the three determinations (other than the first and the subsequent disregarded ones) shall be taken as the representative value of electric strength for the test sample material.

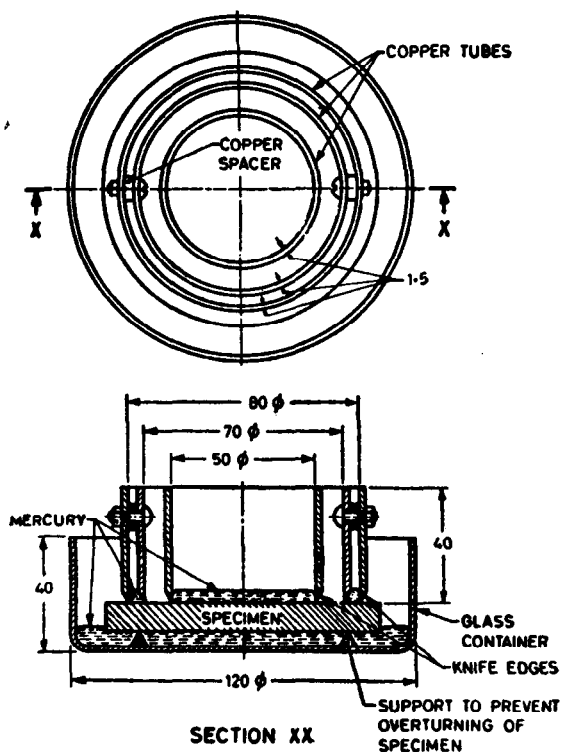
20. DETERMINATION OF SURFACE RESISTIVITY AFTER IMMERSION IN WATER

20.1 Carry out the determination on at least two test specimens prepared from the same test sample material.

20.2 Apparatus — Electrodes as shown in Fig. 12 and electric circuit as shown in Fig. 13 are suitable for measuring surface resistivity.

20.3 Preparation of Test Specimens — The test specimens shall be prepared as prescribed in 19.3 and 19.3.1.

20.4 Conditioning — Immediately after the specimen has cooled to room temperature immerse it for 24.0 ± 0.5 hours in water at a temperature of $27^\circ \pm 2^\circ\text{C}$. Take out the specimens from water and remove all surface moisture by pressing with dry blotting or filter paper.



All dimensions in millimetres.

FIG. 12 DETAILS OF ELECTRODES FOR SURFACE AND VOLUME RESISTIVITY TESTS

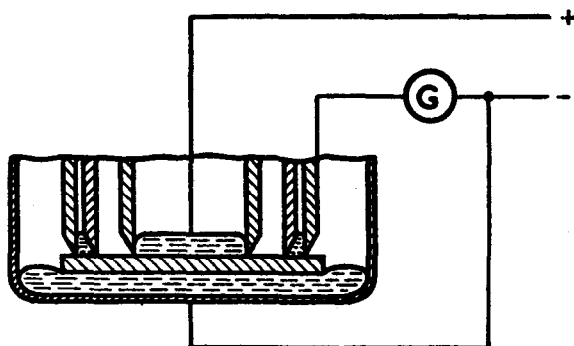


FIG. 13 CIRCUIT FOR SURFACE RESISTIVITY TEST

20.5 Procedure — Measure the surface resistance of each specimen at $27^{\circ} \pm 5^{\circ}\text{C}$ in 10 to 15 minutes from the time of its removal from water. The relative humidity of the laboratory atmosphere at the time of test shall not exceed 75 percent. The surface resistance between the electrodes shall be measured one minute after the application of electrical pressure at 500 ± 50 volts dc.

20.6 Calculation

$$\text{Surface resistivity (ohms)} = \log_{10} \left\{ \frac{2\pi S}{\log_e \left(\frac{D}{d} \right)} \right\}$$

where

S = surface resistance in ohms,

D = internal diameter in mm of upper outer electrode, and

d = internal diameter in mm of upper inner electrode.

NOTE — For the apparatus shown in Fig. 12, surface resistivity $\log_{10}$ ohms = $\log_{10} 18.7 S$.

20.7 Report — The mean of the two determinations shall be taken as the representative value of surface resistivity for the test sample material.

21. DETERMINATION OF VOLUME RESISTIVITY

21.1 Carry out the determination on at least two test specimens prepared from the same test sample material.

21.2 Apparatus — Electrodes shown in Fig. 12 and electric circuit shown in Fig. 14 are suitable for measuring volume resistivity.

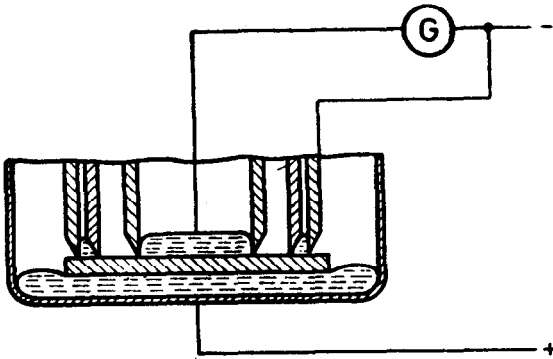


FIG. 14 CIRCUIT FOR VOLUME RESISTIVITY TEST

21.3 Preparation of Test Specimens — The test specimens shall be moulded discs not less than 100 mm (or 4.00 in) in diameter and 3.0 ± 0.2 mm (or 0.125 ± 0.010 in) thick. The specimens shall be flat.

21.3.1 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²) and a mould temperature of 155° to 170° C. Do not pre-heat the moulding material before loading into the mould and do not allow the mould to breathe. Do not allow the curing time to exceed 6 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the mouldings. Cool the specimens to room temperature and test within 24 hours of moulding.

21.4 Procedure — Measure the volume resistance of each specimen at $27^\circ \pm 5^\circ\text{C}$ between the electrodes, one minute after the application of electrical pressure at a potential difference of 500 ± 50 volts DC.

21.5 Calculation

$$\text{Volume resistivity (log}_{10}\text{ohm.cm)} = \log_{10} \frac{A R}{t}$$

where

A = area in cm² of upper electrode (19.6 cm² of upper electrode shown in Fig. 12),

R = resistance in ohms between the upper and the lower electrodes, and

t = thickness in cm of the specimen.

21.6 Report — The mean of the two determinations shall be taken as the representative value of volume resistivity for the test sample material.

22. DETERMINATION OF POWER FACTOR AND PERMITTIVITY AT A FREQUENCY OF ONE MEGACYCLE PER SECOND

22.1 Carry out the determination on at least two test specimens prepared from the same test sample material.

22.2 Preparation of Test Specimens — The specimens shall be moulded discs 50 ± 1 mm (or 2.00 ± 0.04 in) in diameter and 1.5 to 3.2 mm (or 1/16 to 1/8 in) thick.

22.2.1 The specimens shall be flat and the thickness at any point shall not vary from the average thickness by an amount exceeding 0.025 mm (or 0.001 in). It is permissible to grind or otherwise machine the specimen in order to comply with these requirements.

22.2.2 Mould the specimens in a flash, a positive or a semi-positive mould, with a moulding pressure of 150 to 450 kg/cm² (or 1 to 3 ton/in²) and a mould temperature of 155° to 170°C. If desired, pre-heat the material before loading into the mould. Do not allow the curing time to exceed 5 minutes; but less time may be used provided the specimens are properly cured. If found necessary, chill the mould before ejection of the moulding. Cool to room temperature in a desiccator. Test the specimens within 24 hours of moulding.

22.3 Procedure — Measure the power factor and permittivity of the specimens at $27^\circ \pm 5^\circ\text{C}$ by the method prescribed for determination of power factor and permittivity in IS : 1998-1962 Methods of Test for Thermosetting Synthetic Resin Bonded Laminated Sheets or by any other method which gives the same results.

22.4 Report — The mean of the two determinations of power factor and permittivity shall be taken as the representative values for power factor and permittivity for the test sample material.

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